Effect of Sulfates on the Properties of Terrazzo Tiles

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Abstract

An experimental study was conducted to investigate the effect of total sulfates in the mix on the properties of terrazzo tiles . These properties are modulus of rupture , ultrasonic pulse velocity , surface absorption and total absorption .Four levels of total SO_3 content in the mix were investigated , namely 4 , 6 , 8 and 10 % by weight of cement .

Test results indicate that there is an optimum SO_3 content in which the ultrasonic pulse velocity is maximum and total absorption is minimum for all ages considered in this research .This optimum SO_3 content was found to be 6 % by weight of cement giving increase in ultrasonic pulse velocity ranging from 4.65 to 13.96 % and a decrease in total absorption from 0.163 to 3.17 % . On the other hand , increasing the percentage of SO_3 has the effect of decreasing the modulus of rupture .For example , at the 6 % sulfate content , the decrease in modulus of rupture is varied from 12.7 to 39 % .

The analysis of the test results also shows that the properties of terrazzo tiles are deteriorated by increasing the period of curing for mixes of high SO_3 content .

يتضمن هذا البحث دراسة مختبريه حول تأثير نسبة الأملاح الكبريتية الكلية قي الخلطة على خواص الكاشي الموزائيك والتي تشمل معاير التصدع وسرعة الأمواج فوق الصوتية وامتصاص الوجه والامتصاص الكلي. تم استعمال نسب مختلفة من الكبريتات وهي 4 و 6 و 8 و 10 % من وزن السمنت .

أشارت نتائج الفحوصات إلى وجود نسبة مثلى للكبريتات يتم الحصول فيها على اكبر سرعة للموجات فوق الصوتية واقل نسبة للامتصاص الكلي ولكل الأعمار التي تم تناولها في هذا البحث . هذه النسبة المثلى هي 6% من وزن السمنت معطية زيادة في سرعة الأمواج فوق الصوتية تتراوح من 4.65 الى 13.96 % ونقصان في الامتصاص الكلي بين 0.163 و 3.17 % . ومن جهة أخرى فان زيادة نسبة الكبريتات يؤدي إلى نقصان معاير التصدع ،وكمثال على ذلك فان هذا النقصان يتراوح بين 12.7 و 39 % عندما يكون محتوى الكبريتات هو 6 % .

لقد بين تحليل نتائج الفحوصات ألمختبريه بان خواص الكاشي الموزائيك تتضرر بزيادة فترة الإنضاج وذلك للخلطات الحاوية على نسبة عالية من الكبريتات .

Introduction

Most of the concrete aggregates used in Iraq have a comparatively high amount of sulfates . The sulfates in aggregate are generally composed of magnesium , sodium , and calcium sulfates . Calcium sulfate is the most common salt present in Iraqi aggregate since it represents about 95 % of overall sulfates [Raouf 1970].

Sulfates can react with different hydration products, mainly calcium aluminate hydrate and $Ca(OH)_2$ resulting in the formation of calcium sulfoalominate and gypsum respectively . This reaction, known as sulfate attack , is companied by a considerable increase in solid volumes , so it leads to expansion , cracking and loss of concrete strength .

Sulfate attack may be internal or external according to the source of sulfate salts . In internal attack the sulfates come from ingredients of concrete , whereas the basis of sulfates in external attack is ground water or soil .

The reaction of the sulfates with hardened cement paste are as follows [Neville 1995];

$$Ca(OH)_2 + Na_2SO_4 . 10\ H_2O \longrightarrow CaSO_4 . 2H_2O + 2NaOH + 8H_2O$$

$$3CaO.Al_2O_3 + 3CaSO_4 .2H_2O + 25\ H2O \longrightarrow 3CaO.Al_2O_3 .3CaSO_4 . 31H_2O$$

Many investigators such as [Al- Rawi 1977, Al-Zayori 1988, Al- Rawi and Abdul-Latif 1998 , Tailor 1999, Alwash 2005] have studied the influence of sulfate attack . They concluded that the effect of sulfates on various concrete properties depends mainly on type and content of sulfates .

Failure of concrete structures in the Middle East is primarily caused by the contamination of sand with sulfates in the form of gypsum [Al-Rawi et al. 2001] . This problem is likely to happen for other materials that have similar ingredients to concrete such as terrazzo tiles .

In the present research , the effect of a calcium sulfate on the properties of terrazzo tiles is investigated . The modulus of rupture , surface absorption , total absorption , and ultrasonic pulse velocity tests were the investigated properties .

Experimental work

Materials

Cement

Two types of cement were used , sulfate resisting Portland cement (S.R.P.C.) manufactured by Karbala cement plant , and white Portland cement (W.P.C) . They conformed to Iraqi specification No. 5 / 1984 . The properties of these cements are presented in Table 1 .

Fine aggregate (sand)

Al-Akhaidhur well-graded sand was used. The physical and chemical properties are listed in Table 2. Its grading conformed to Iraqi specification No.45 / 1984, Zone 2.

Coarse aggregate (gravel)

The coarse aggregate with a maximum size of 14 mm was used. Table 3 shows the properties of the gravel.

Table 1 : Properties of the cements

Oxide	%		IQS 5 -1984 Limits
	S.R.P.C.	W.P.C.	
CaO	64.94	61.0	----
SiO_2	22.1	22.42	----
MgO	1.45	1.5	----
Fe_2O_3	5.12	0.56	----
Al_2O_3	3.3	4.4	----
SO_3	2.17	2.7	< =2.5 if C3A < 5% < =2.8 if C3A > 5%
Free Lime	1.78	1.7	----
L.O.I	0.63	3	<=4%
I.R	1.26	1.2	<= 1.5%
Compound composition according to Bogue's equations			
C_3S	54.14	32.6	----
C_2S	22.6	39.7	-----
C_3A	0.09	10.7	< =3.5 % for S.R.P.C
C_4AF	15.56	1.7	-----
L.S.F	0.89	0.9	0.66 -1.02
Physical properties of the cements			
Test	Test result		IQS 5-1984 Limits
	S.R.P.C.	W.P.C.	
Fineness, (Blaine) , m^2/kg	280	320	> = 250 for S.R.P.C. > = 230 for W.P.C .
Soundness(Autoclave) , %	0.28	0.3	< = 0.8
Initial setting time , min.	125	130	> = 45 min
Final setting time , min.	230	250	< = 600 min
Comp. strength at 3days ,MPa	18	16	> = 15
Comp. strength at7days , MPa	26	24	> = 23

Table 2 : Properties of the fine aggregate (Sand)

Sieve Size (mm)	Percentage passing	IQS 45-1984 Limits Zone 2
10	100	100
4.75	95	90 -100
2.36	85	75 – 100
1.18	70	55 - 90
0.6	55	35 - 59
0.3	27	8 – 30
0.15	5	0 - 10
Properties	**Test results**	**IQS 45-1984 Limits**
Sulfate content (SO_3) , %	0.5	<= 0.5
Fineness modulus	2.6	----
Materials finer than sieve No. 200 , %	5	<= 5

Table 3 : Properties of the coarse aggregate (gravel)

Sieve Size (mm)	Percentage passing	IQS 45-1984 Limits Size (5-14) mm
19	100	100
14	95	90 -100
9.5	75	50 – 85
4.75	5	0 - 10
Properties	**Test results**	**IQS 45-1984 Limits**
Sulfate content (SO_3) , %	0.2	<= 0.1
Specific gravity	2.6	----
Bulk density ,Kg/m^3	1650	----
Materials finer than sieve No. 200 , %	5	<= 5

Gypsum

The added gypsum is a natural gypsum rock brought from Al- Muthana cement factory . Its gradation was finer than the sand. The SO_3 content in the gypsum was 37% .

Terrazzo stones

Terrazzo stones get from Galala region with a maximum size of 20 mm were used in the preparation of the face layer of terrazzo tiles.

Limestone dust

Limestone dust was added to the mixture of the face layer of tiles . Table 4 illustrates the chemical and physical properties of the limestone dust.

Table 4 : Properties 0f the limestone dust

Oxide	%
CaO	50.3
SiO_2	6
MgO	0.5
Fe_2O_3	0.5
Al_2O_3	2
SO_3	0.2
L.O.I	40.5
Physical properties of the limestone dust	
Sieve Size (mm)	**Percentage passing**
0.6	100
0.3	85
0.15	20
0.075	5

Procedures

Mix proportions

Two types of mixes were used . The proportions by weight were 1 : 1 : 1 : 0.7 (W. P.C. : limestone dust : stones : water) for the face layer and 1 : 1.5 : 0.5 : 0.3 (S.R.P.C : sand : gravel : water) for the bottom layer of terrazzo tiles .

The level of total SO_3 in the mixture of bottom layer was changed by adding the gypsum to give level of total SO3 of (6 , 8 ,10) percent by weight of cement .

Mixing , casting , and compaction

The W.P.C , limestone dust and the necessary added water were manually mixed together and poured into the steel mould with dimensions(300 * 300 * 30 mm) .This mix represents a first layer of terrazzo tile . The S.R.P.C. , sand , and the required amount of gypsum were mixed dry . Then the water was added until a homogeneous mix was obtained . After that , the mix was put on top of the first layer and compacted using a hydraulic machine with a maximum load of (5 ton). The mixing and casting of tiles were carried out in Al- Bayati factory for tiles production.

Curing and age of testing

Immediately after compaction, the specimen was demoulded carefully and left to dry indoors at the room temperature . Some of the specimens were stored in tap water tank until testing age of 14 and 28 days . Other specimens were immersed in water for 28 days and then subjected to different cyclic wetting and drying , namely one to six cycles of 7 days wet and 7 days dry exposure . These specimens were tested at ages 56 , 84 and112 days .

Testing methods

Modulus of rupture test

One point load flexure test was performed on three samples of tiles (300 * 300 * 30 mm). The modulus of rupture test was carried out according to Iraqi specification No. 1042 / 1984

Ultrasonic pulse velocity test

The velocity of the ultrasonic pulse for bottom layers of tiles was determined according to B.S.1881:Part 203 . Portable ultrasonic concrete tester was used for this purpose . A thin layer of grease was applied to act as a couplant . The path length of pulse traveling was measured and the time of its traveling was recorded by using the method of surface waves transmission . The ultrasonic pulse velocity (U.P.V.) was determined as follows :

$$\mathbf{U.P.V. = L / T}$$

Where :

L : Path length (km) , T : Transit time (sec) .

Total and surface absorption tests

The total and surface absorption were determined according to the procedure outlined in Iraqi specification No. 1042 / 1984 .The total absorption was measured by drying a specimen to a constant mass , immersing it in water for 24 hours , and calculating the increase in mass as a percentage of dry mass .In the case of surface absorption , the surface layer was only immersed in water to the depth of 5 mm for 24 hours , and the increase in mass per unit area of the specimen was measured .

Results and discussion

Modulus of rupture test results

Figure 1 shows the effect of total SO_3 in the mix on the modulus of rupture of the terrazzo tile specimens . It can be seen from the figure that , increasing the amount of total SO_3 in the mix reduces the modulus of rupture for all ages .For example the decrease in modulus of rupture is ranged from 12.7 to 39 % for the mixes of 6% SO_3 level . This could be attributed to the formation of calcium sulfoaluminate (Ettringite) witch associated with the increase in volume of the reactant . This volume change leads to internal stresses and cracking . In addition , the consequence of internal sulfate attack also include loss of strength due to the loss of cohesion in hydrated cement paste and adhesion between it and aggregate particle[Neville 1995]

The results of modulus of rupture as a function of curing time are plotted in Figure 2 . It can be observed that the modulus of rupture decreases with time for mixes of 6 , 8 and 10 % SO_3 levels , whereas the reference mix exhibits continuing enhancement with age . The reason of such behavior is that , the mixes of high sulfate content suffer from severe attack due to the presence of water during curing period , which increases the proceeding of sulfate reactions .

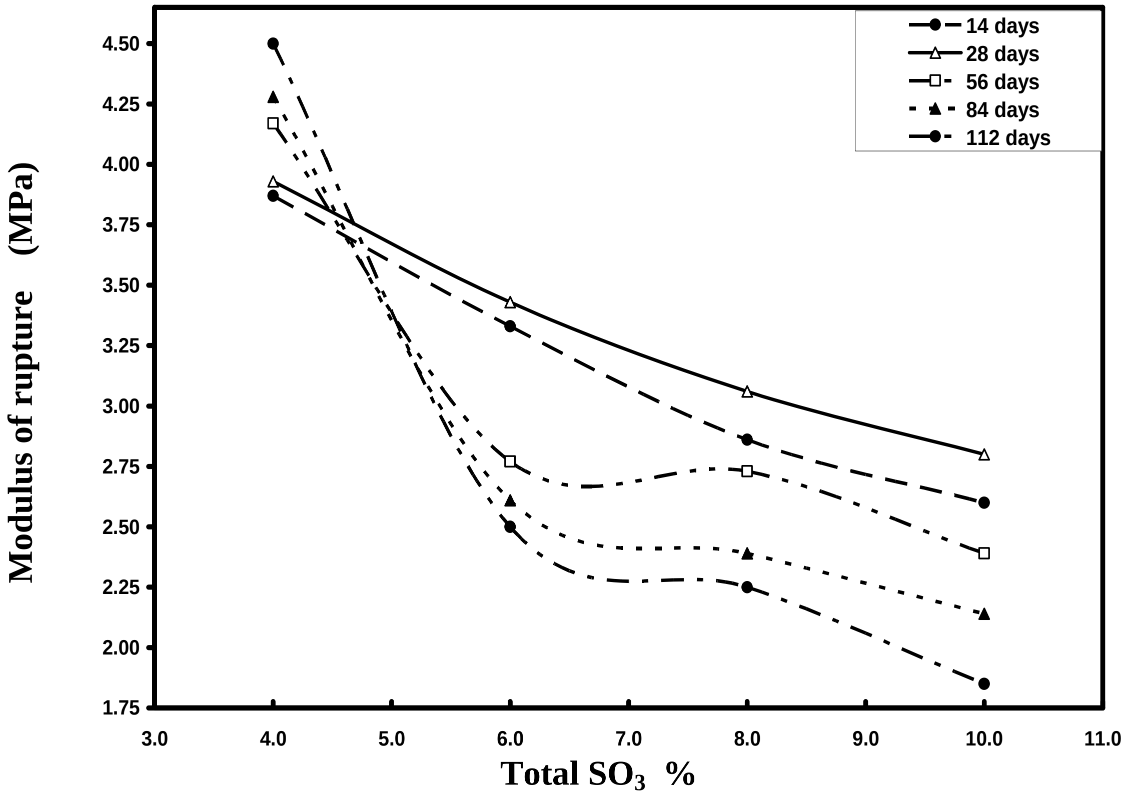


Figure 1: Effect of total SO_3 content on the modulus of rupture for various ages

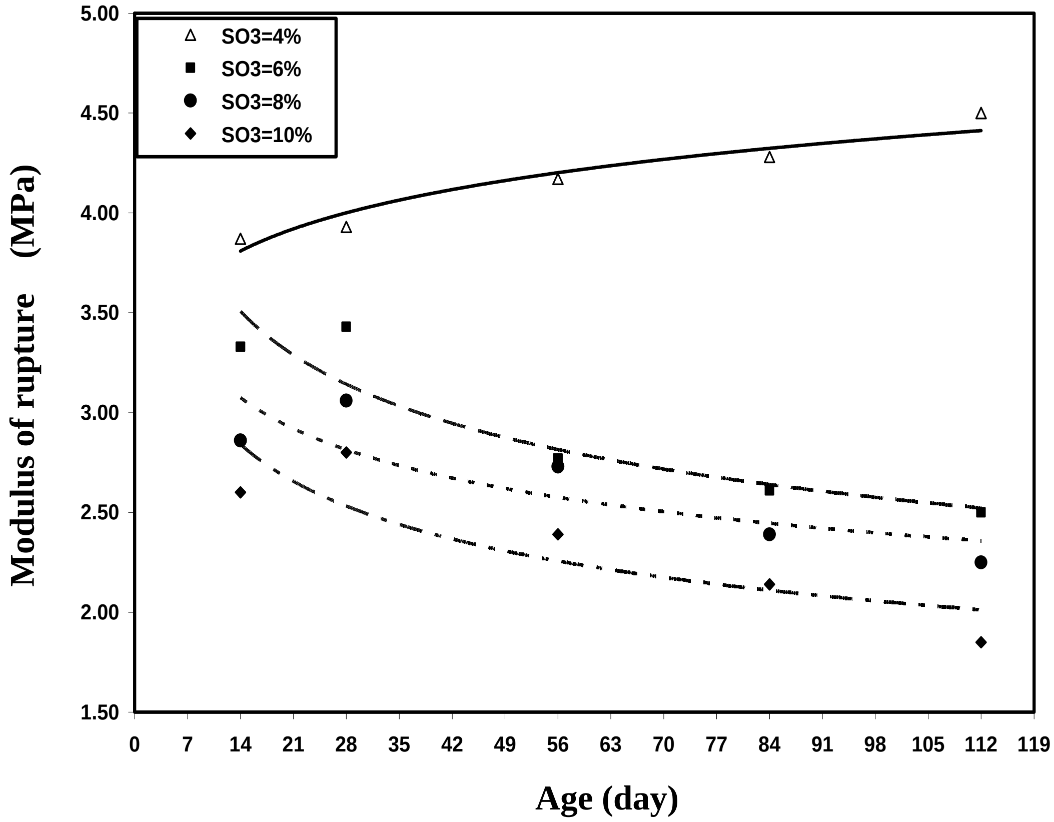


Figure 2: The Modulus of rupture versus age for different total SO_3 content

Ultrasonic pulse velocity test results

The measured pulse velocity of a material depends on density and the elastic properties of the material [Jones 1962] . Generally , high pulse velocity readings are indication of good quality material .

The variation of the values of pulse velocity of the bottom layer of tiles with the total SO_3 content in the mix are shown in Figure3. It is clear from the figure that the ultrasonic pulse velocity increases as the sulfate content is increased up to an optimum SO_3 content beyond witch the improvement achieved in velocity goes down. This optimum content is 6 % by weight of cement in which the increase in pulse velocity is ranged from 4.65 to 13.96 % . The explanation may be that , the pores in the back layer of tile may be filled by the Ettringite . This leads to increase density and hence ultrasonic pulse velocity . On the other hand , as the SO_3 content is increased beyond the optimum , the pulse velocity starts to decrease because of the formation of further amount of Ettringite which is accompanied by expansion and cracking .

Figure 4 illustrates the effect of curing age on the ultrasonic pulse velocity of tiles . Again , as with the modulus of rupture the figure indicates that there is a decrease in velocity as the age of curing is increased for the mixes of high sulfate content .

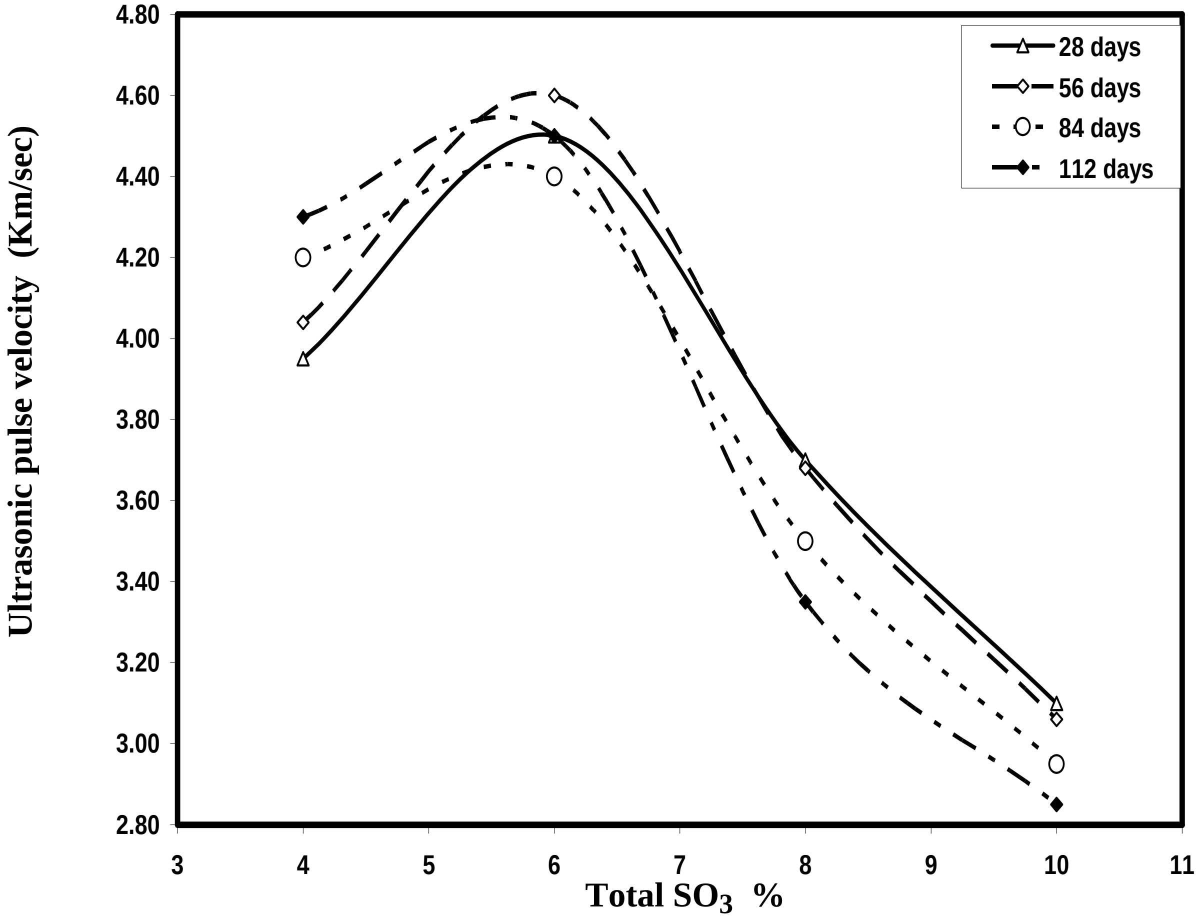


Figure 3: Effect of total SO_3 content on the ultrasonic pulse velocity for various ages

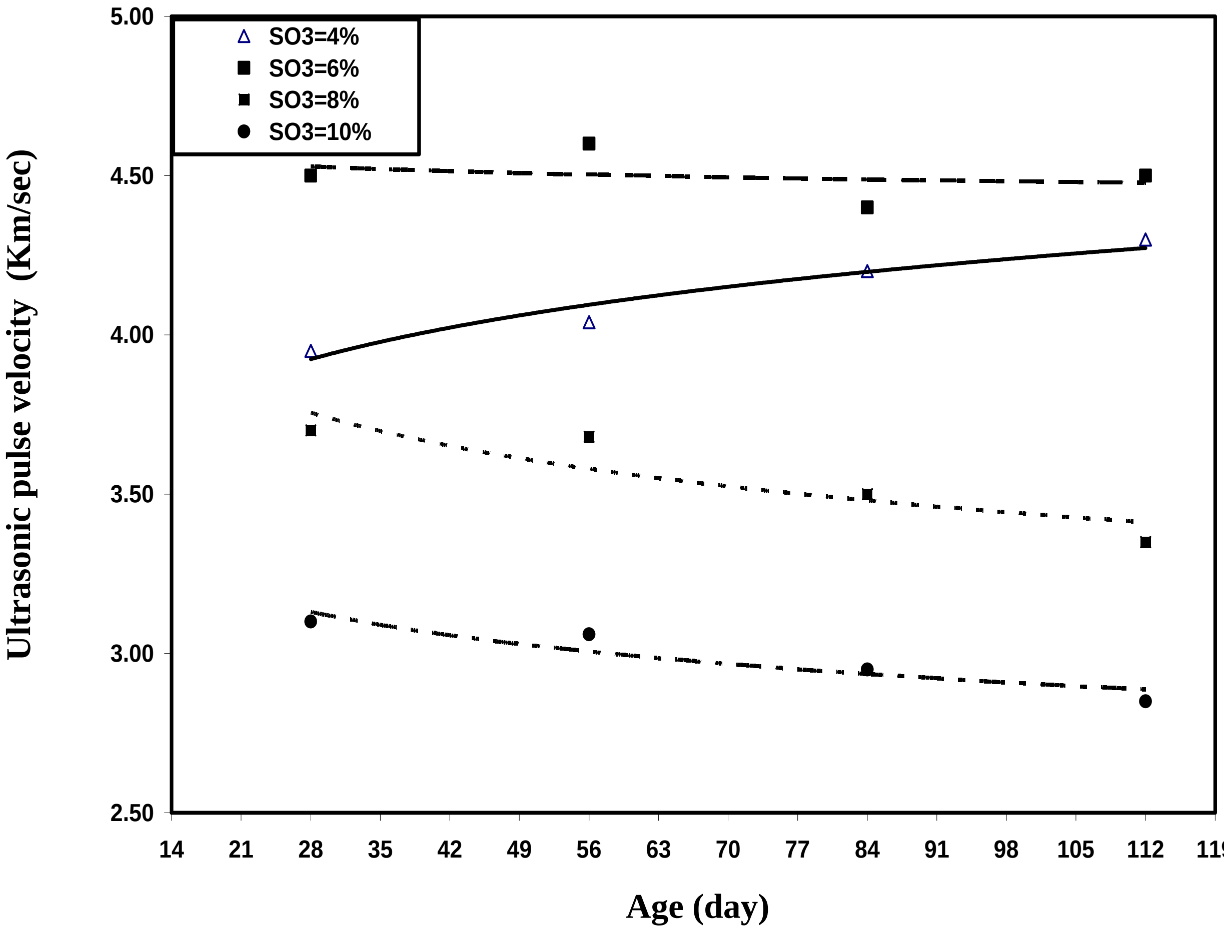


Figure 4: The ultrasonic pulse velocity versus age for different total SO_3 content

Total and surface absorption test results

The relation between total absorption of terrazzo tile specimens and the sulfate content is shown in Figure 5 . The figure shows that the total absorption tends to decrease with increasing sulfate content up to an optimum amount . Further increase in the sulfate content beyond the optimum causes an increase in total absorption . As with the ultrasonic pulse velocity , the 6 % SO_3 level in the mix is the optimum giving the decrease in total absorption ranges from 0.163 to 3.17 % . This is because that the Ettringite may gradually fill the pores witch are responsible for absorption .However , total absorption of tiles is adversely affected by increasing the SO_3 content beyond the optimum . This can be attributed to the excessive expansion and even visible cracking of hardened cement paste due to the effect of severe sulfate attack . Figure 6 illustrates the variation of total absorption of tiles with time . The figure shows the increase in total absorption when increasing the curing age for the mixes of 6 , 8 and 10 % sulfate content .

The data obtained from the surface absorption test are plotted in Figures 7 and 8 . These figures indicate that this property is not affected by SO_3 content in the mix . It can also be seen that the surface absorption decreases with increasing curing period due to the progress of hydration process for white Portland cement witch leads to decrease porosity .

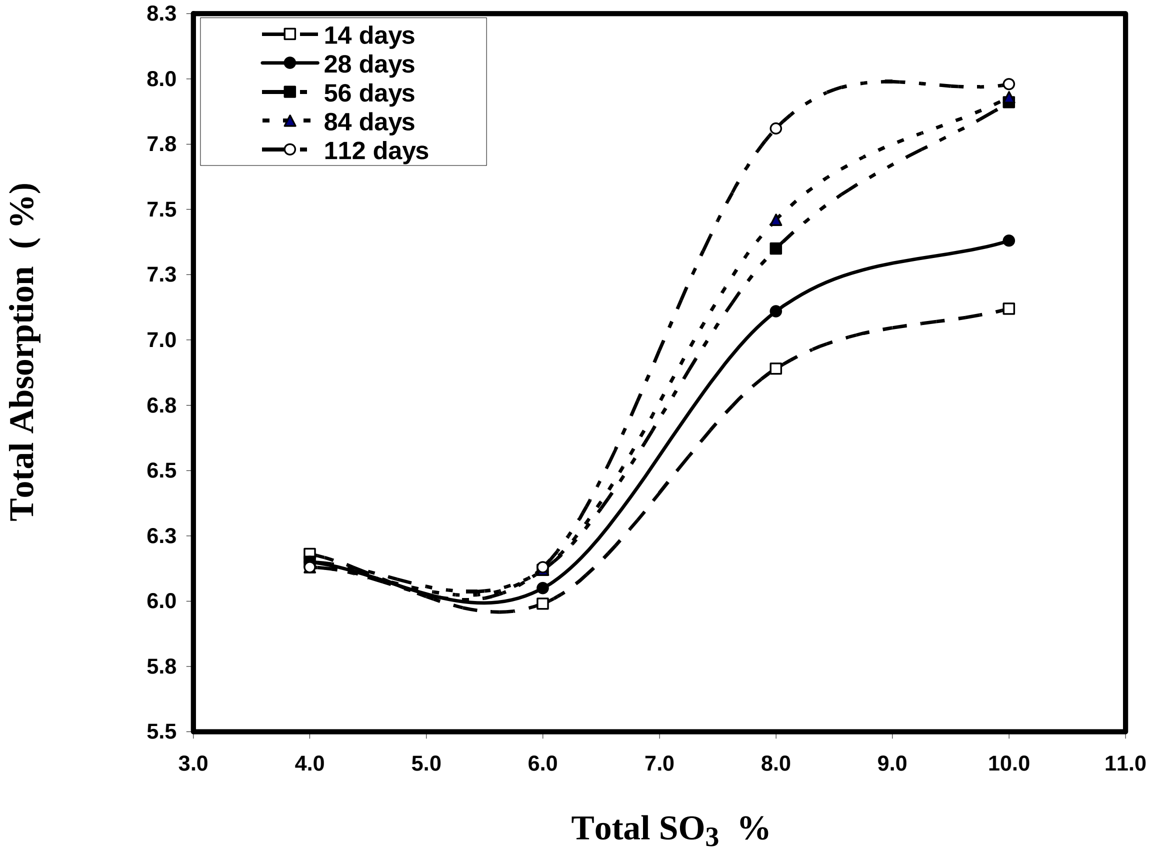


Figure 5: Effect of total SO_3 content on the total absorption for various ages

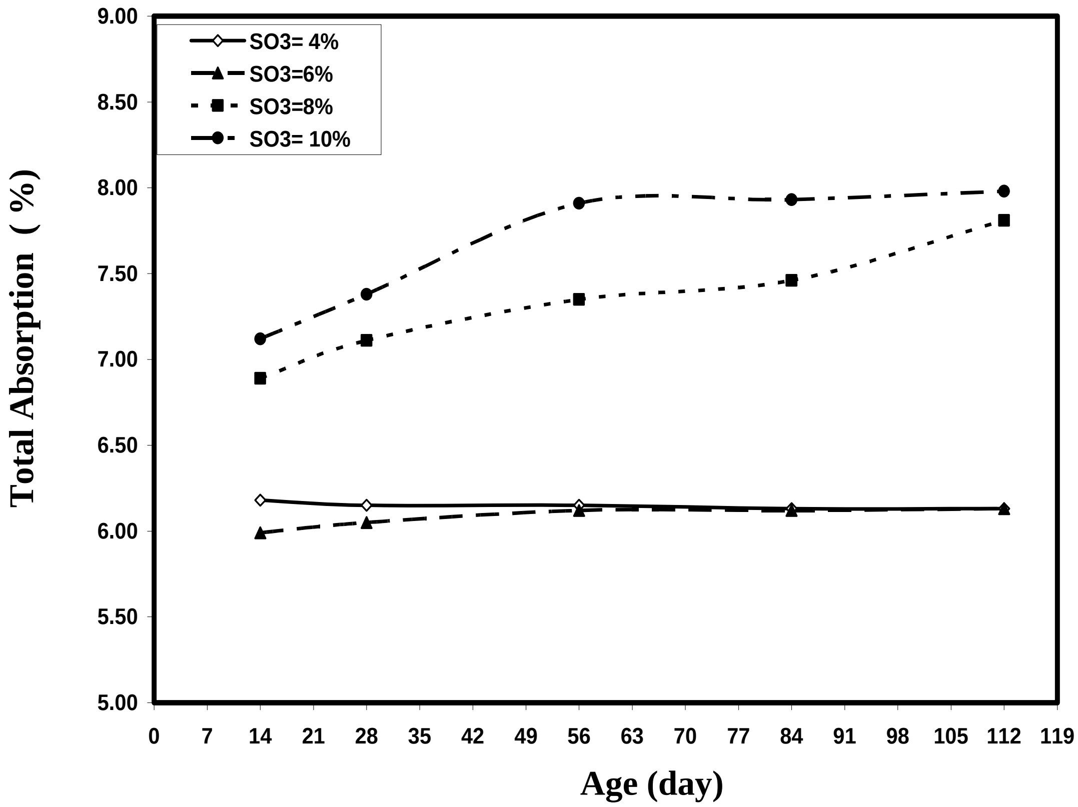


Figure 6: The total Absorption versus age for different total SO_3 content

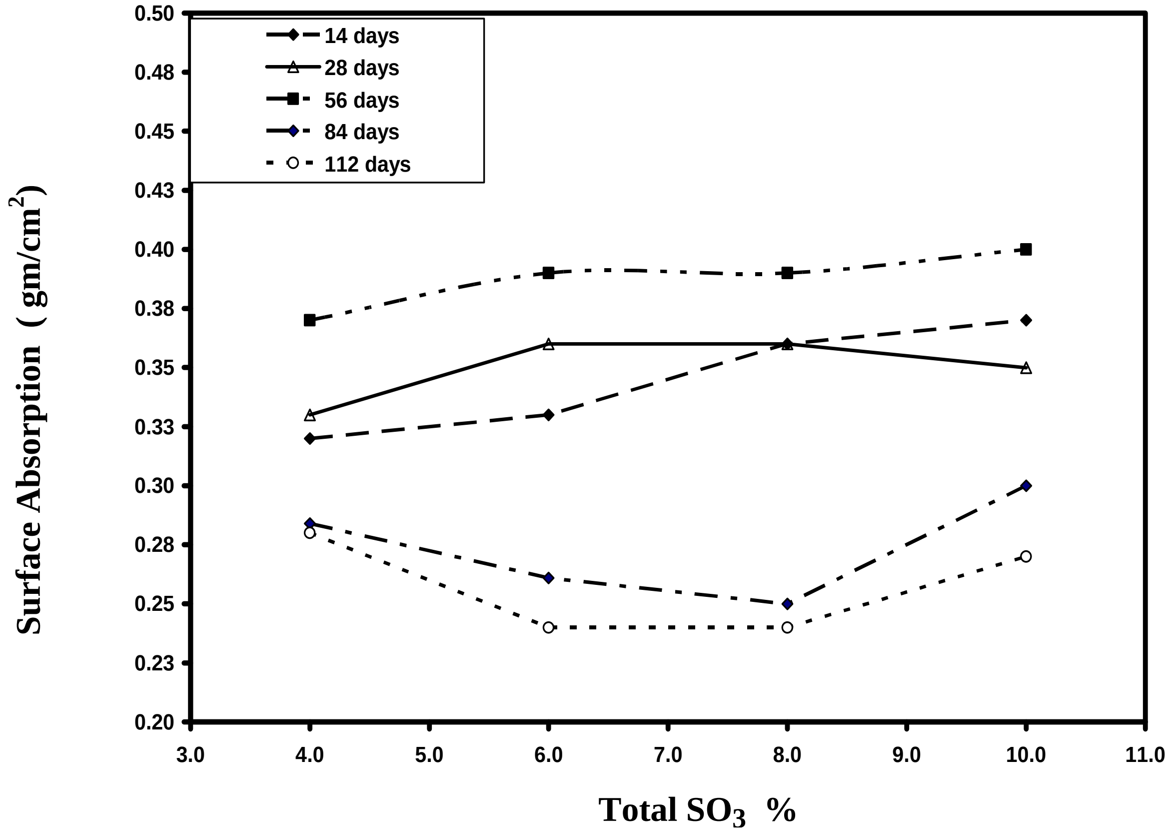


Figure 7: Effect of total SO_3 content on the Surface absorption for various ages

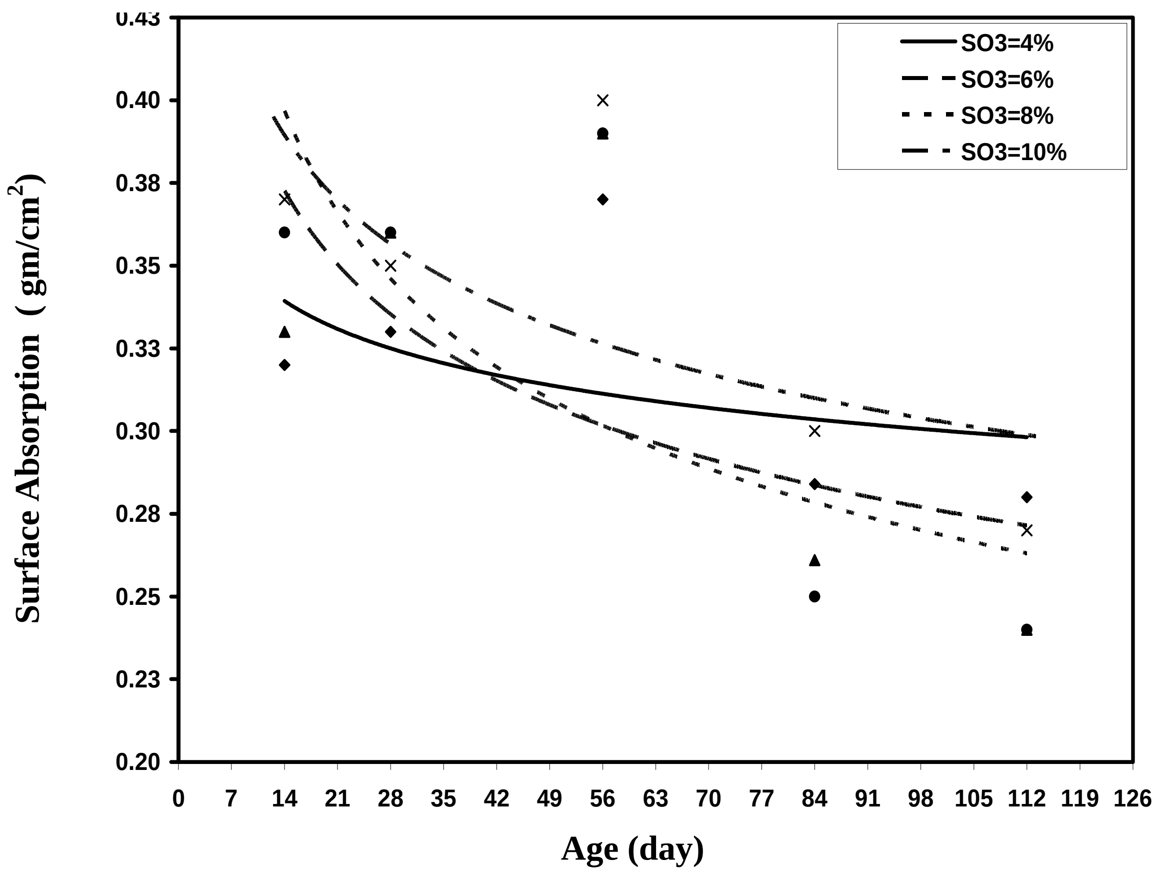


Figure 8: The total absorption versus age for different total SO_3 content

Conclusions

Based on the experimental results of the present research , the following conclusions can be drown :

1- It was found that , there appears to be an optimum sulfate content beyond witch the total absorption of terrazzo tiles starts to increase and ultrasonic pulse velocity decrease . A total SO_3 content in the mix of 6 % by weight of cement is such an optimum content . At this percentage , the maximum improvement in ultrasonic pulse velocity and total absorption is 13.96 % and 3.17 % respectively .

2- The results indicated that increasing the amount of sulfates leads to decrease modulus of rupture of terrazzo tile specimens .For example , at 6 % sulfate content the decrease in modulus of rupture is ranged from 12.7% to 39 % .

3- It was found that increasing the curing period has a bad effect on the modulus of rupture , ultrasonic pulse velocity and total absorption of mixes with 6 , 8 and 10 % SO_3 levels .

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